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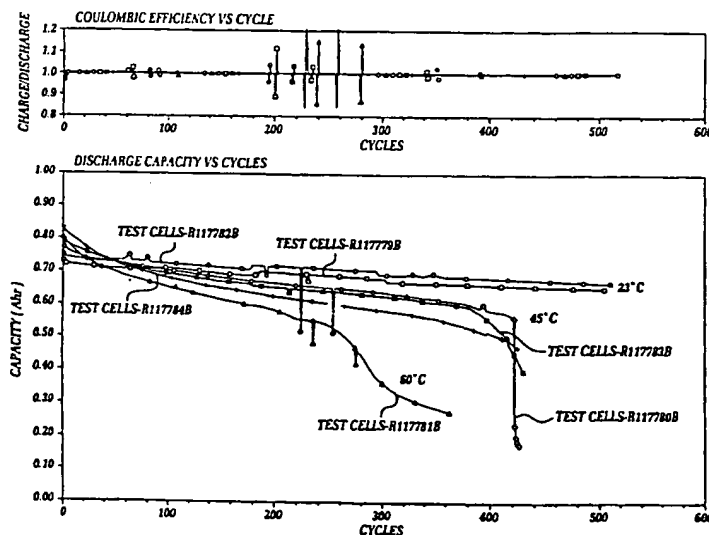
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(54) Title: **ACTIVE MATERIAL HAVING EXTENDED CYCLE LIFE**



(57) Abstract: In one embodiment, the invention provides a novel composition which is stabilized against decomposition when used as an active material for an electrochemical cell. The active material of the present invention comprises particles of spinel lithium manganese oxide (LMO) enriched with lithium by a decomposition product of lithium hydroxide forming a part of each of the LMO particles. The spinel LMO product formed by the decomposition of lithium hydroxide in the presence of the LMO is characterized by a reduced surface area and increased capacity retention (reduced capacity fading) as compared to the initial, non-treated, non-enriched spinel. In another aspect, the treated spinel LMO product is combined with lithium carbonate in a cathode mixture.

## ACTIVE MATERIAL HAVING EXTENDED CYCLE LIFE

### Field of the Invention

This invention relates to electrochemical cells  
5 and batteries, and more particularly, to such cells and  
batteries having lithium-based active material.

### Background of the Invention

Lithium batteries are prepared from one or more  
lithium electrochemical cells. Such cells have included  
10 an anode (negative electrode), a cathode (positive  
electrode), and an electrolyte interposed between  
electrically insulated, spaced apart positive and  
negative electrodes. The electrolyte typically comprises  
a salt of lithium dissolved in one or more solvents,  
15 typically nonaqueous (aprotic) organic solvents. By  
convention, during discharge of the cell, the negative  
electrode of the cell is defined as the anode. During use  
of the cell, lithium ions ( $\text{Li}^+$ ) are transferred to the  
negative electrode on charging. During discharge,  
20 lithium ions ( $\text{Li}^+$ ) are transferred from the negative  
electrode (anode) to the positive electrode (cathode).  
Upon subsequent charge and discharge, the lithium ions  
( $\text{Li}^+$ ) are transported between the electrodes. Cells  
having metallic lithium anode and metal chalcogenide  
25 cathode are charged in an initial condition. During  
discharge, lithium ions from the metallic anode pass  
through the liquid electrolyte to the electrochemically  
active material of the cathode whereupon electrical  
energy is released. During charging, the flow of lithium

loss in capacity. That is, the initial capacity available (amp hours/gram) from  $\text{LiMn}_2\text{O}_4$ ,  $\text{LiNiO}_2$ , and  $\text{LiCoO}_2$  is less than the theoretical capacity because less than 1 atomic unit of lithium engages in the electrochemical reaction. Such an initial capacity value is significantly diminished during the first cycle operation and such capacity further diminishes on every successive cycle of operation. The specific capacity for  $\text{LiMn}_2\text{O}_4$  is at best 148 milliamp hours per gram. As described by those skilled in the field, the observed reversible capacity is on the order of 60% of the aforesaid value. Obviously, there is a tremendous difference between the theoretical capacity (assuming all lithium is extracted from  $\text{LiMn}_2\text{O}_4$ ) and the actual capacity when much less than one atomic unit of lithium is extracted as observed during operation of a cell. For the metal oxides listed above, only about 0.5 atomic units of lithium is reversibly cycled during cell operation. Thus, the presently known and commonly used, alkali transition metal oxide compounds suffer from relatively low capacity. Therefore, there remains the difficulty of obtaining a lithium- containing chalcogenide electrode material having acceptable capacity without disadvantage of significant capacity fading (loss) when used in a cell.

Capacity fading is well known and is calculated according to the equation given below. The equation is used to calculate the first cycle capacity loss. This same equation is also used to calculate subsequent progressive capacity loss during subsequent cycling relative back to the first cycle capacity charge reference.

Summary of the Invention

In one embodiment, the invention provides a novel composition which is stabilized against decomposition when used as an active material for an electrochemical cell. Problems observed with degradation and decomposition of active material have been described in co-pending applications Serial No. 09/307,335, filed May 7, 1999, and in then co-pending PCT/US97/22525, filed November 21, 1997, and in then co-pending U.S. Serial No. 08/762,081, filed December 9, 1996, now U.S. Patent No. 5,869,207, in the names of J. Barker, Y. Saidi, and C. S. Kelley and assigned to the assignee of the present invention. The active material of the present invention comprises particles of spinel lithium manganese oxide (LMO) enriched with lithium by a decomposition product of a lithium-containing compound. This lithium-containing compound meets three criteria: (1) it is lithium-containing; (2) decomposable at low enough temperature to release Li and not cause oxygen deficiency from the LMO; and (3) does not contain potentially harmful contaminants to the LMO performance (e.g., transition metals). Desirable compounds are, for example, lithium acetate ( $\text{LiC}_2\text{H}_3\text{O}_2$ ), lithium oxalate ( $\text{Li}_2\text{C}_2\text{O}_4$ ), lithium formate ( $\text{LiOOCH}$ ), and lithium tartrate ( $\text{Li}_2\text{C}_4\text{H}_4\text{O}_6$ ). The most preferred compound is lithium hydroxide. The decomposition product forms a part of each of the LMO particles. The spinel LMO product formed by the decomposition of lithium containing compound in the presence of the LMO is characterized by a reduced surface area and increased capacity retention (reduced capacity fading) as compared to the initial, non-treated, non-enriched spinel.

of, and preferably all of, the lithium hydroxide in the presence of the lithium manganese oxide. Depending on the temperature selected, a portion of the lithium hydroxide is decomposed or reacted.

5                   The result is a treated spinel lithium manganese oxide  $\text{Li}_{1+y}\text{Mn}_{2-y}\text{O}_4$  characterized by reduced surface area and increased lithium content as compared to an untreated spinel lithium manganese oxide  $\text{Li}_{1+x}\text{Mn}_{2-x}\text{O}_4$  where  $y$  is greater than  $x$ .

10                   In one aspect, the aforesaid heating to decompose the lithium hydroxide is conducted in a static (i.e., non-flowing) air atmosphere or in a flowing air atmosphere. In another alternative, it is conducted in inert (e.g., nitrogen) atmosphere since the LMO would not  
15                   lose oxygen at the temperature required for the Li incorporation reaction (e.g., 400°C). The heating is conducted for a time of up to about 5 hours and the amount of lithium hydroxide contained in the mixture, is up to about 10%, and desirably up to about 5% by weight  
20                   of the total mixture. This weight percent is based on the lithium hydroxide being first air dried since it is somewhat hygroscopic and it is preferred to evaporate any water. Preferably, the amount of anhydrous lithium hydroxide in the mixture is on the order of 1.2 weight  
25                   percent with the balance being the LMO. The process of the invention can utilize either anhydrous  $\text{LiOH}$ , or  $\text{LiOH} \cdot \text{H}_2\text{O}$  (monohydrate). The monohydrate is the stable hydrate formed. If using the monohydrate, obviously a change has to be made to the weight of reactant used to compensate  
30                   for the  $\text{H}_2\text{O}$  content.

### Brief Description of the Drawings

**Figure 1** is a two-part graph showing the results of testing cells, rocking chair batteries, having an anode comprising MCMB active material cycled with a counter-electrode comprising treated lithium manganese oxide active material as per the Examples presented herein. **Figure 1A** is Coulombic Efficiency and **1B** is Discharged Capacity, each versus Cycles. The cells were cycled between about 3 and 4.2 volts for up to 500 cycles. The cells were cycled at 23°C, 45°C, and 60°C, with a two hour discharge rate (C/2) and a two hour charge rate (C/2); and an additional potentiostatic period at 4.2 volts until current drops to 10% of the C/2 rate. The geometric surface area of the positive electrode was about 180 square centimeters.

**Figure 2** contains data for another group of cells similar to **Figure 1**, except that in these cells the positive electrode also contained particles of lithium carbonate along with the treated lithium manganese oxide. All other conditions were the same.

**Figure 3** is a two-part graph showing the results of testing another group of cells, rocking chair batteries, having an anode (negative electrode) comprising MCMB active material cycled with a positive electrode comprising treated lithium manganese oxide active material as per the Examples presented herein. **Figure 3A** is Coulombic Efficiency and **3B** is Discharged Capacity, each versus Cycles. The cells were cycled between about 3 and 4.2 volts for up to 500 cycles. The cells were cycled at 23°C, 45°C, and 60°C, with a two hour discharge rate (C/2) and a two hour charge rate

Detailed Description of the Preferred Embodiments

In one aspect of the invention, treated lithium manganese oxide is obtained essentially as a result of the thermal decomposition of a lithium containing compound on the lithium manganese oxide. Preferably, this is accomplished by heating a mixture of lithium manganese oxide and lithium hydroxide at an elevated temperature. It is believed that the treated lithium manganese oxide made in accordance with the invention differs fundamentally from lithium manganese oxide known in the art. This difference is reflected in the treated lithium manganese oxide's distinguished electrochemical performance in a cell and also distinguished by the process by which the treated lithium manganese oxide is prepared.

Before further describing the invention, it is useful to understand the characteristics of untreated lithium manganese oxide. The nominal general formula  $\text{LiMn}_2\text{O}_4$  represents a narrow range of spinel lithium manganese oxide compounds (referred to as LMO) which have stoichiometry that varies somewhat in the relative proportion of lithium, manganese and oxygen, but still having the spinel structure. Oxygen deficient spinels are not favored here. The invention provides relatively lithium-rich spinels. This is in contrast to relatively lithium deficient spinels of the formula  $\text{Li}_{1-x}\text{Mn}_2\text{O}_4$  which are not favored here. An example of such lithium deficient spinels may be found in U.S. Patent No. 5,443,929. In contrast to '929, and according to the invention, the lithium manganese oxide material before treatment is represented by  $\text{Li}_{1+x}\text{Mn}_{2-x}\text{O}_4$  where  $0 < x \leq 0.08$ . Preferably,  $x$  is 0.02 to 0.08 before treatment.

a suitable solvent. Thereafter, the solvent is removed from the resulting mixture by heating, simple evaporation or other equivalent means known in the art. Suitable solvents include water or alcohol, such as methanol, ethanol, and the like.

Lithium hydroxide used to treat the spinel is known to be hygroscopic. This means that about one atomic unit of water will accompany each atomic unit of lithium hydroxide. Therefore, it is preferred to pre-dry the lithium hydroxide in air at a temperature of about 200°C for up to about sixteen hours. This yields an essentially anhydrous form of lithium hydroxide which is most preferred for the method of the invention.

In the above-described mixture containing the preferred lithium hydroxide and the LMO, the amount of lithium hydroxide is desirably from 0.1 to 10 percent by weight, more desirably from 0.5 to 5, and preferably 1.2 percent by weight of the mixture, with the LMO constituting the balance.

The prepared mixture containing the lithium hydroxide (LiOH) and the LMO is subjected to heating. This heating step is carried out at a temperature high enough to initiate the decomposition of the lithium hydroxide on the surface of the individual particles of the LMO. Preferably, the temperature is high enough to essentially completely decompose the lithium hydroxide, leaving behind a lithium-enriched spinel. The heating temperature is below the temperature at which the LMO will be decomposed and the heating temperature is below the melting point of the LMO. Importantly, the temperature is also below the temperature where LMO would



Typical cell configurations will be described with reference to **Figures 6** and **7**.

A typical laminated battery cell structure 10 is depicted in **Figure 6**. It comprises a negative electrode side 12, a positive electrode side 14, and an electrolyte/separator 16 there between. Negative electrode side 12 includes current collector 18, and positive electrode side 14 includes current collector 22. A copper collector foil 18, preferably in the form of an open mesh grid, upon which is laid a negative electrode membrane 20 comprising an intercalation material such as carbon or graphite or low-voltage lithium insertion compound, dispersed in a polymeric binder matrix. An electrolyte separator film 16 membrane of plasticized copolymer is positioned upon the electrode element and is covered with a positive electrode membrane 24 comprising a composition of a finely divided lithium intercalation compound in a polymeric binder matrix. An aluminum collector foil or grid 22 completes the assembly. Protective bagging material 40 covers the cell and prevents infiltration of air and moisture.

In another embodiment, a multicell battery configuration as per **Figure 7** is prepared with copper current collector 51, negative electrode 53, electrolyte/separator 55, positive electrode 57, and aluminum current collector 59. Tabs 52 and 58 of the current collector elements form respective terminals for the battery structure.

The relative weight proportions of the components of the positive electrode are generally: 50-90% by weight active material; 5-30% carbon black as the

120-160°C. Subsequent to lamination, the battery cell material may be stored either with the retained plasticizer or as a dry sheet after extraction of the plasticizer with a selective low-boiling point solvent. The plasticizer extraction solvent is not critical, and methanol or ether are often used.

Separator membrane element 16 is generally polymeric and prepared from a composition comprising a copolymer. A preferred composition is the 75 to 92% vinylidene fluoride with 8 to 25% hexafluoropropylene copolymer (available commercially from Atochem North America as Kynar FLEX) and an organic solvent plasticizer. Such a copolymer composition is also preferred for the preparation of the electrode membrane elements, since subsequent laminate interface compatibility is ensured. The plasticizing solvent may be one of the various organic compounds commonly used as solvents for electrolyte salts, e.g., propylene carbonate or ethylene carbonate, as well as mixtures of these compounds. Higher-boiling plasticizer compounds such as dibutyl phthalate, dimethyl phthalate, diethyl phthalate, and tris butoxyethyl phosphate are particularly suitable. Inorganic filler adjuncts, such as fumed alumina or silanized fumed silica, may be used to enhance the physical strength and melt viscosity of a separator membrane and, in some compositions, to increase the subsequent level of electrolyte solution absorption.

In the construction of a lithium-ion battery, a current collector layer of aluminum foil or grid is overlaid with a positive electrode film, or membrane, separately prepared as a coated layer of a dispersion of intercalation electrode composition. This is typically

preferred. The polyethylene oxide of 5,411,820 is an example. More modern examples are the VdF:HFP polymeric matrix. Examples of casting, lamination and formation of cells using VdF:HFP are as described in U.S. Patent Nos. 5,418,091; 5,460,904; 5,456,000; and 5,540,741; assigned to Bell Communications Research, each of which is incorporated herein by reference in its entirety.

As described earlier, the electrochemical cell which utilizes the novel active material of the invention may be prepared in a variety of ways. In one embodiment, the negative electrode may be metallic lithium. In more desirable embodiments, the negative electrode is an intercalation active material, such as, metal oxides and graphite. When a metal oxide active material is used, the components of the electrode are the metal oxide, electrically conductive carbon, and binder, in proportions similar to that described above for the positive electrode. In a preferred embodiment, the negative electrode active material is graphite particles. For test purposes, test cells are often fabricated using lithium metal electrodes. When forming cells for use as batteries, it is preferred to use an intercalation metal oxide positive electrode and a graphitic carbon negative electrode. In the examples below, all test cells contained graphitic carbon active materials. None of the carbon anodes were pre-lithiated prior to assembly in a cell. Various methods for fabricating electrochemical cells and batteries and for forming electrode components are described herein. The invention is not, however, limited by any particular fabrication method.

as a solvent. The preferred plasticizer was dibutyl phthalate (DPB). The slurry was cast onto glass and a free-standing electrode was formed as the solvent was evaporated. The cathode electrode composition was approximately as follows on a percent basis: 72.8 treated LMO; 3.0 carbon; 7.5 binder; and 16.7 plasticizer. In a preferred method according to the invention, prior to cell construction the plasticizer is extracted. Thus, on this basis the proportions are as follows on a percent basis: 87.4 treated LMO; 3.6 Super P carbon; and 9.0 Kynar binder.

#### Example II

Another cathode electrode was fabricated using the treated LMO of the above Example I and along with a lithium carbonate ( $\text{Li}_2\text{CO}_3$ ) additive in particle form. In this case, the cathode was fabricated as described above except that lithium carbonate was added to the formulation which was cast to form the electrode. In this case, the cathode electrode coating composition was approximately as follows on a percent basis: 72.6 treated LMO; 3.0 carbon; 7.5 binder; 0.3  $\text{Li}_2\text{CO}_3$ ; and 16.7 plasticizer. On an extracted basis, this corresponds to 87.0 treated LMO; 3.6 Super P carbon; 0.4  $\text{Li}_2\text{CO}_3$ ; and 9.0 Kynar binder.

A graphite counter electrode was prepared for use with the aforesaid cathode. The graphite counter electrode served as the anode in the electrochemical cell. The graphite electrode was fabricated by solvent casting a slurry of MCMB2528 graphite, binder, plasticizer, and casting solvent. MCMB2528 is a mesocarbon microbead material supplied by Alumina

Table I

	BEFORE	TREATED LMO <sup>1</sup>
Surface Area/m <sup>2</sup> /g	0.85	0.68
d10	10.7	10.4
d50 volume %	31.1	31.0
d97	69.8	68.7
Li content/wt %	4.05	4.34
Lattice Constant a (Å)	8.2158	8.209
Residual LiOH	0	0
Oxygen Deficiency	0	0
Color	Grey/Black	Grey/Black
	<u>x in Li<sub>1+x</sub>Mn<sub>2-x</sub>O<sub>4</sub></u> (from XRD before)	<u>y in Li<sub>1+y</sub>Mn<sub>2-y</sub>O<sub>4</sub></u> (from XRD after)
	0.086	0.120

\*XRD = x-ray diffraction

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<sup>1</sup> Treated LMO prepared by heating 98.8 weight percent LMO and 1.2 wt. % LiOH at 425°C for one hour.

hydroxide and used to form a cathode which further included particles of lithium carbonate as per Example II. **Figure 2A** shows rechargeability and **Figure 2B** shows cyclability and capacity of the cell prepared in accordance with Example II. The capacity was determined at constant current cycling for up to 500 cycles at 23°C, 45°C, and 60°C versus graphite anode at C/2 charge and discharge rates. The results of **Figure 2** demonstrate an improvement over that of **Figure 1**. This indicates that the heat treated LMO, combined in a cathode mixture with lithium carbonate, has dramatically improved results particularly when cycled at 45°C and 60°C. Most of the cells, even at the higher temperatures, were capable of cycling even after 500 cycles. The test was terminated after 500 cycles.

The cells cycled at 23°C maintained over 95% of its capacity to 100 cycles; over 96% of its capacity to 200 cycles; and over 91% of its capacity at about 500 cycles.

The cells cycled at 45°C maintained over 95% of its capacity to 100 cycles; over 90% of its capacity to 200 cycles; and over 79% of its capacity at about 500 cycles. The recharge ratio maintained its value close to one in all cases.

The cells cycled at 60°C maintained 84% capacity after 100 cycles; 79% capacity after 200 cycles, and 67% capacity after 500 cycles.

Additional groups of cells were prepared in accordance with the method of Example I, that is, LMO

30% of their original capacity for up to about 40 cycles. See **Figure 5**, plot (c) labeled untreated LMO.

Further cells were prepared using untreated LMO, that is, LMO in the "before" condition as shown in Table 1, but also including particles of lithium carbonate in the cathode formulation. On the basis of 100 parts by weight of total active material, the particulate lithium carbonate constituted about 1.22 weight percent and the untreated lithium manganese oxide the balance. These cells were only able to cycle up to 50 cycles. At about 50 cycles, the cells retained only about 40% of their original capacity. The cycling conditions for all of the aforesaid comparative cells were at 23°C versus the graphite anode at two hour discharge and five hour charge rate. See **Figure 5**, plot (b) labeled LMO + particulate  $\text{Li}_2\text{CO}_3$ .

Still further cells were prepared with LMO heat treated with lithium carbonate at a temperature of about 500°C and up to 800°C, and typically 600°C to 750°C. This heat treatment is at a temperature much greater than the 300°C to up to 500°C, and typically 400°C, used with the lithium containing compound of the present invention. The results for cells prepared with lithium manganese oxide heat treated with lithium carbonate are labeled as plot (a) in **Figure 5**. These cells lost more capacity than the cells heat treated with the preferred lithium hydroxide of the invention.

Based on the comparative cells, there is a dramatic difference and improvement when lithium manganese oxide is heat treated with lithium hydroxide.

**Claims:**

1. A method of treating spinel lithium manganese oxide particles which comprises the steps of (a) forming a mixture comprising said lithium manganese oxide particles and lithium hydroxide; and (b) heating said mixture for a time and at a temperature sufficient to decompose said lithium hydroxide, and to provide treated spinel lithium manganese oxide characterized by reduced surface area and increased lithium content as compared to untreated spinel lithium manganese oxide.
2. The method of claim 1 wherein the untreated spinel lithium manganese oxide of step (a) is represented by the formula  $\text{Li}_{1+x}\text{Mn}_{2-x}\text{O}_4$ ; and wherein said treated spinel having increased lithium content is represented by the formula  $\text{Li}_{1+y}\text{Mn}_{2-y}\text{O}_4$ , where  $0 < x < y \leq 0.20$ .
3. The method of claim 1 wherein the lithium hydroxide is in particle form in step (a).
4. The method of claim 1 wherein said heating step (b) is conducted in an air atmosphere.
5. The method of claim 1 wherein said heating is conducted at a temperature in a range of about 300 to about 450°C.
6. The method of claim 1 wherein said heating is conducted for a time of about 10 minutes to about 5 hours.



said lithium-rich spinel having the formula  $\text{Li}_{1+y}\text{Mn}_{2-y}\text{O}_4$ ,  $0 < y \leq 0.2$ .

12. The composition of claim 11 wherein said lithium rich spinel is represented by the formula  $\text{Li}_{1+y}\text{Mn}_{2-y}\text{O}_4$  where  $0.08 \leq y \leq 0.20$ .

13. A mixture comprising particles of lithium-rich spinel lithium manganese oxide having the formula  $\text{Li}_{1+y}\text{Mn}_{2-y}\text{O}_4$  where  $0 \leq y \leq 0.20$  and particles of lithium carbonate.

14. The mixture of claim 13 which further includes a binder and particles of conductive carbon, where the binder is intermingled with the spinel particles, the carbonate particles, and the carbon particles.

15. An electrode having an active material and a binder, said active material comprising particles of lithium-rich spinel lithium manganese oxide having within said particles more lithium and less manganese at interstitial sites as compared to a stoichiometric spinel  $\text{LiMn}_2\text{O}_4$ ; said lithium-rich spinel having a formula  $\text{Li}_{1+y}\text{Mn}_{2-y}\text{O}_4$  where  $y$  is greater than zero and less than or equal to 0.20, and a surface area less than  $0.8 \text{ m}^2/\text{g}$  (square meter per gram).

16. The electrode of claim 15 wherein said active material has a discharge capacity per unit weight of over 90milliamp hours per gram after 200 cycles.

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Figure 1A

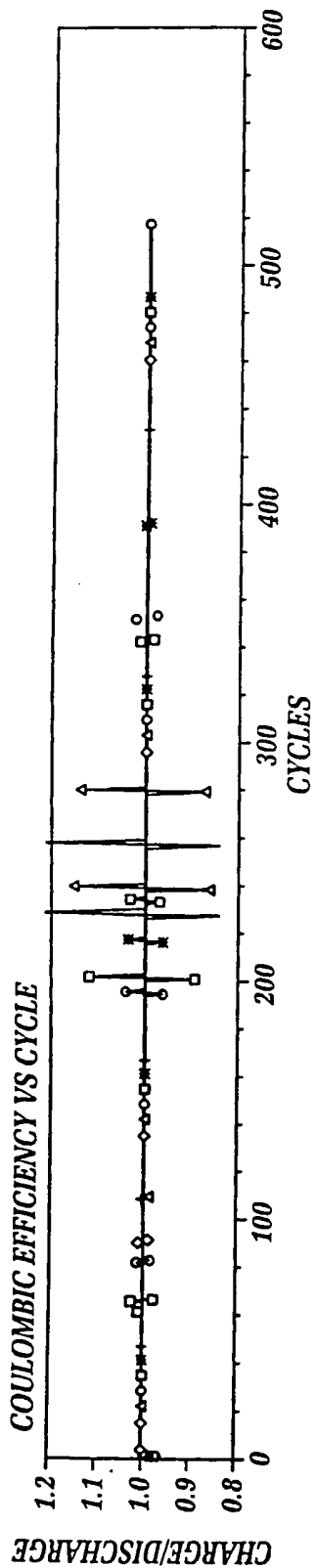


Figure 1B

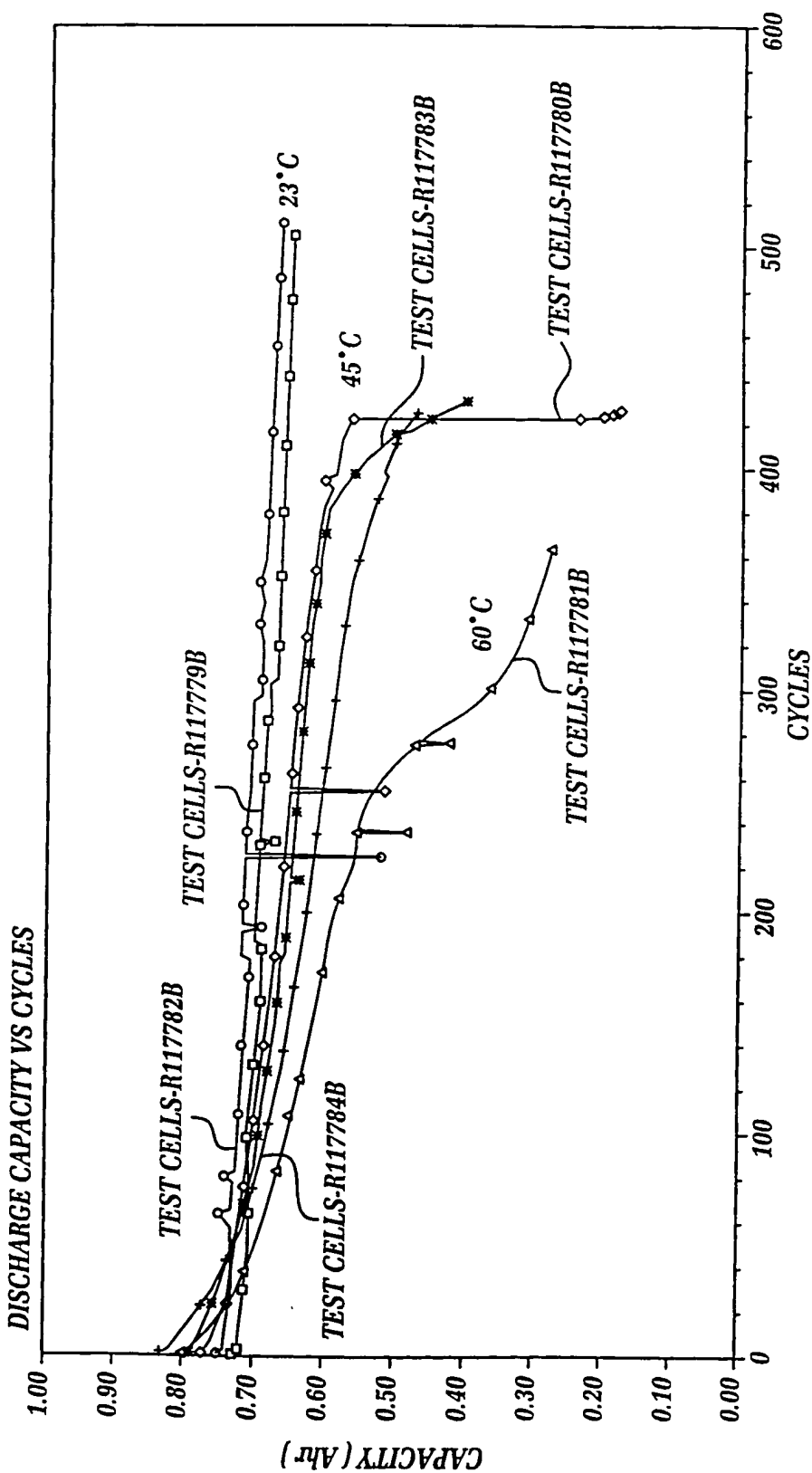


Figure 2A

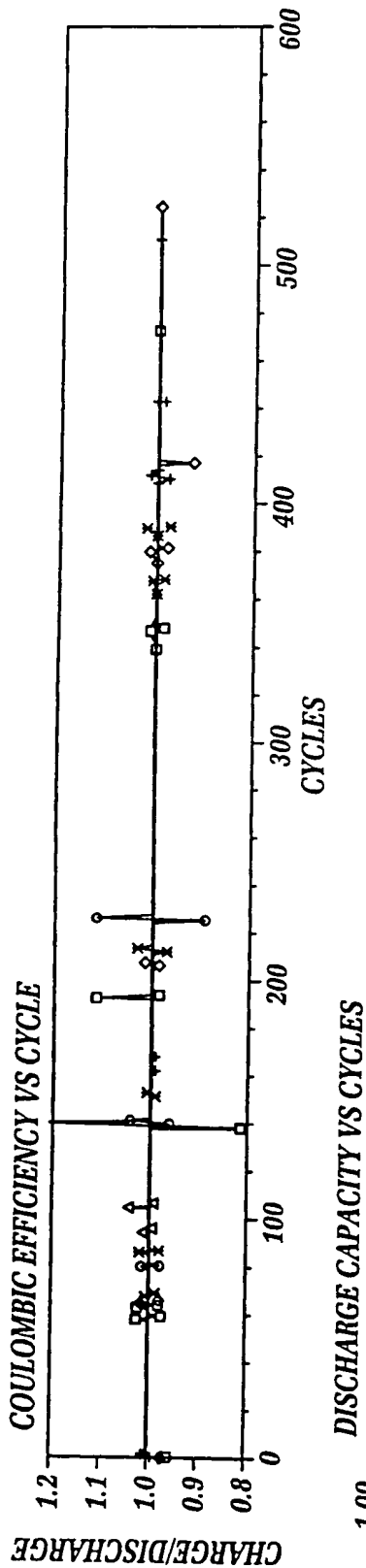


Figure 2B

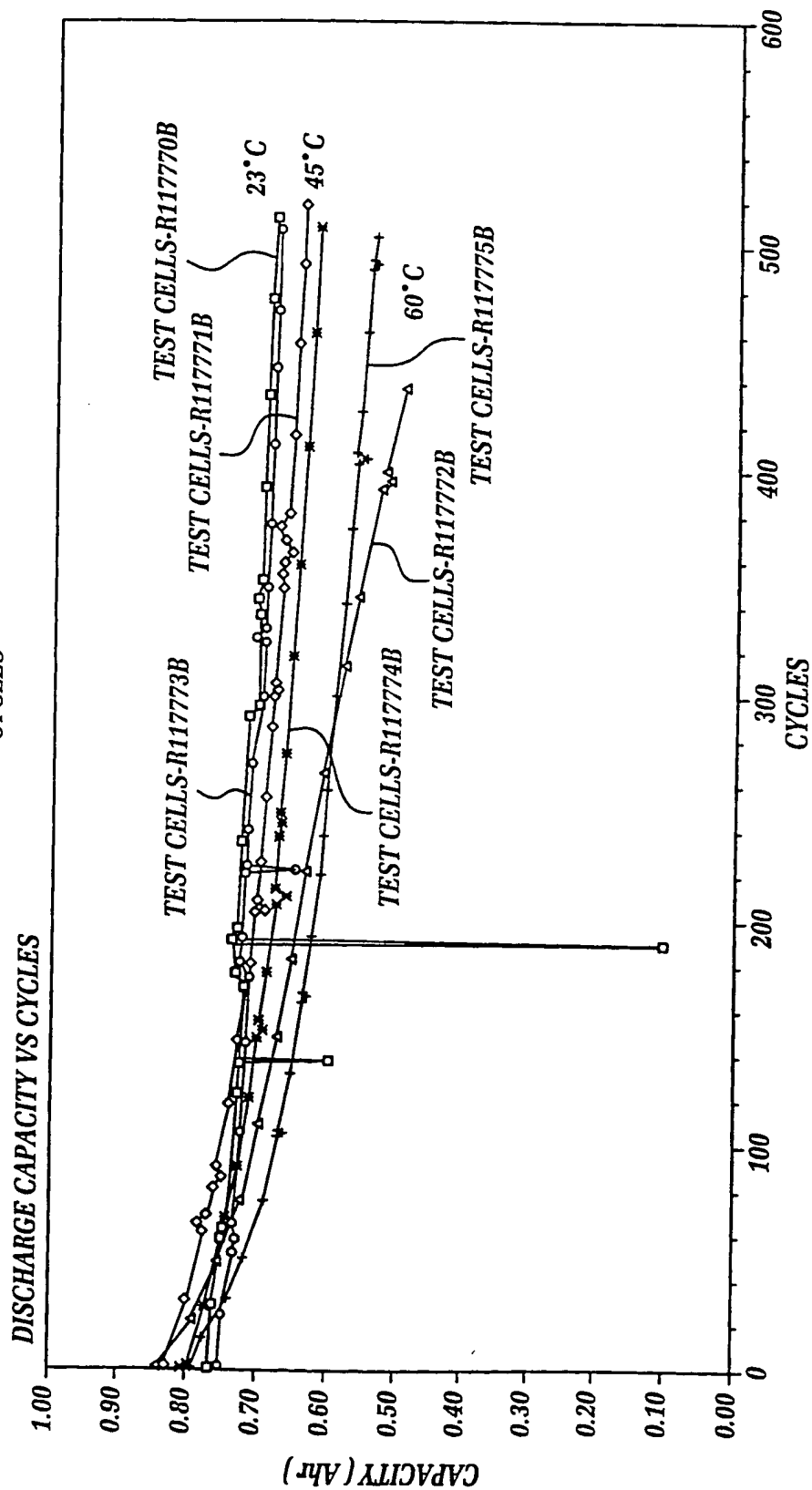


Figure 3A

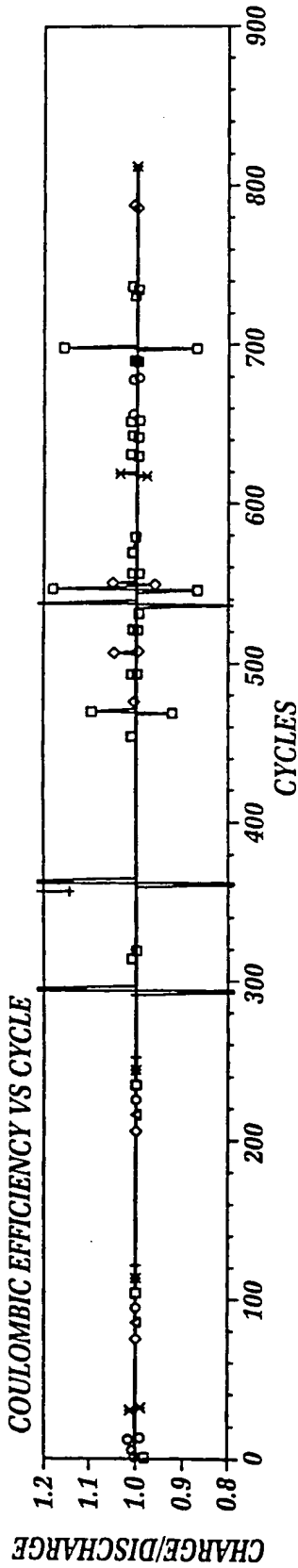


Figure 3B

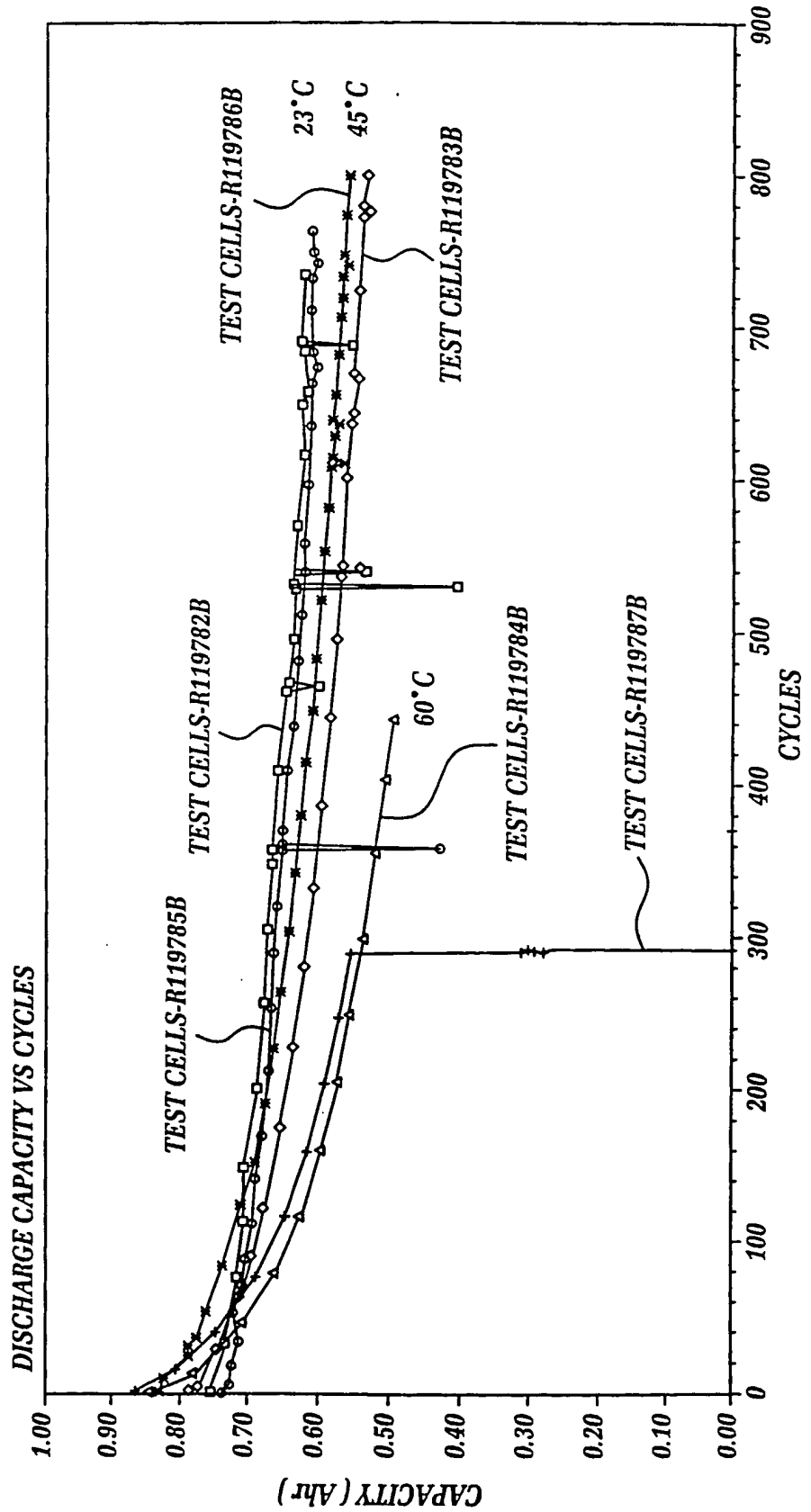


Figure 4A

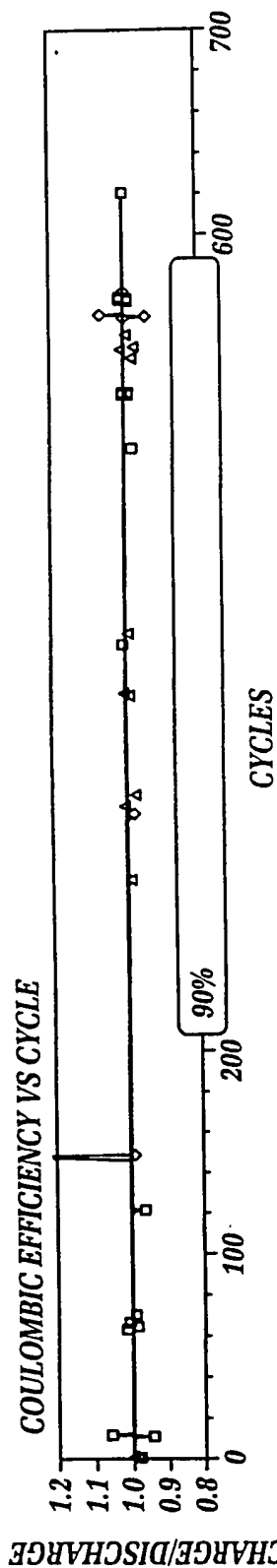
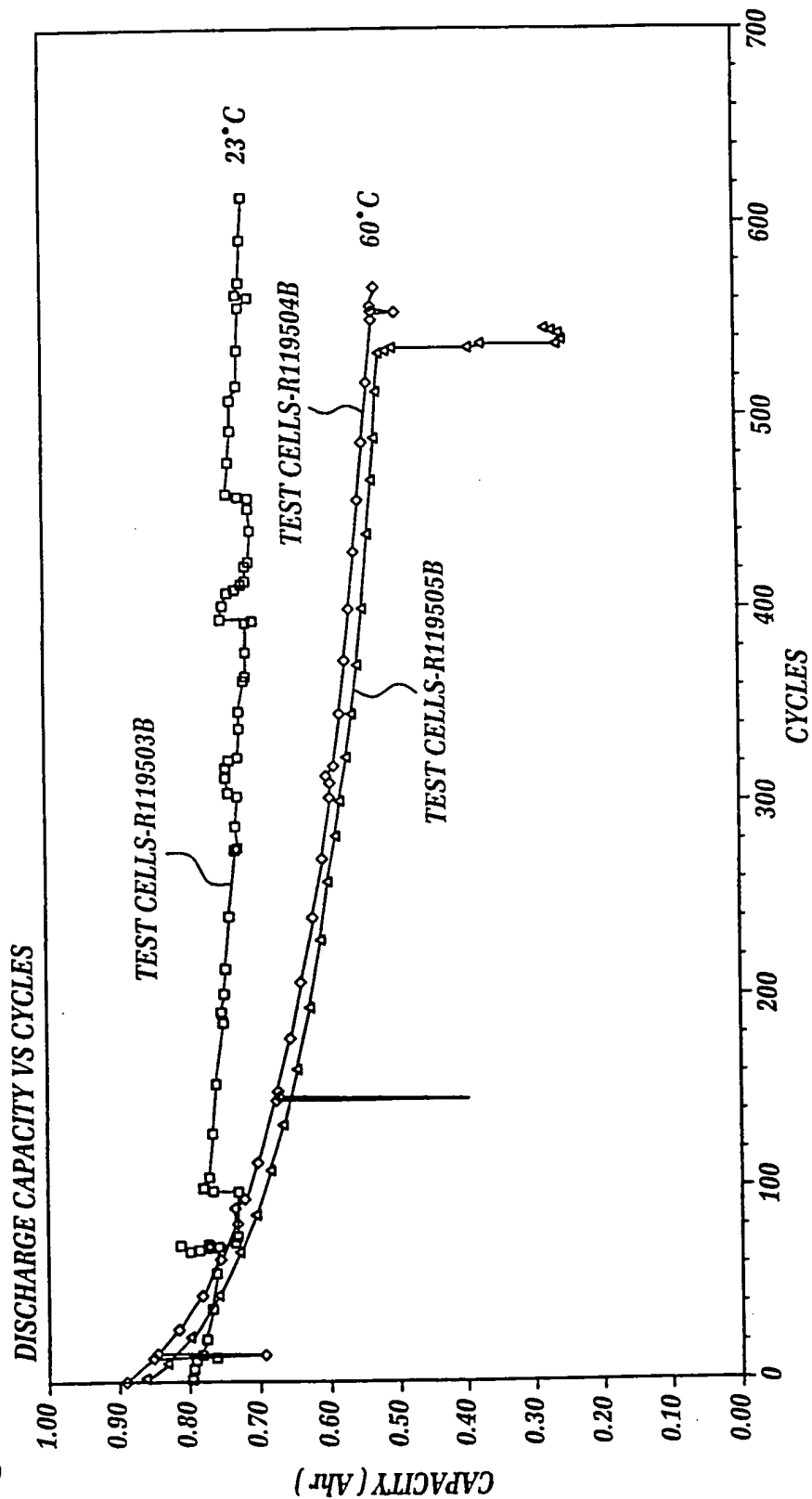


Figure 4B



GRAPHITE/LMO : CELLS CYCLED AT 60°C DISCHARGE = C/2 ; CHARGE = C/5

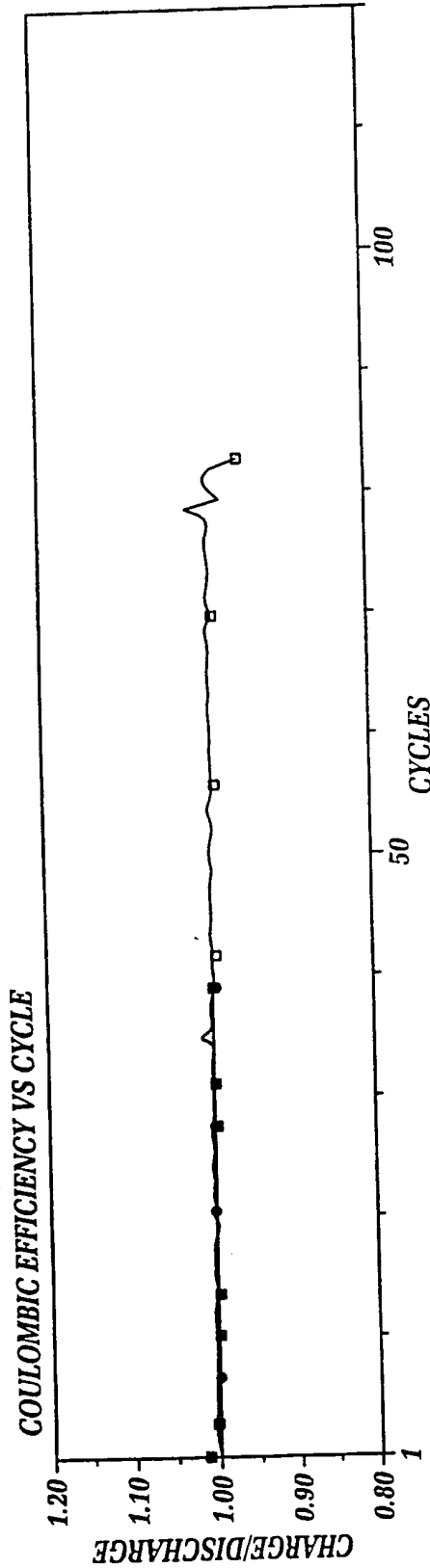


Figure 5A

GRAPHITE/LMO : CELLS CYCLED AT 60°C DISCHARGE = C/2 ; CHARGE = C/5

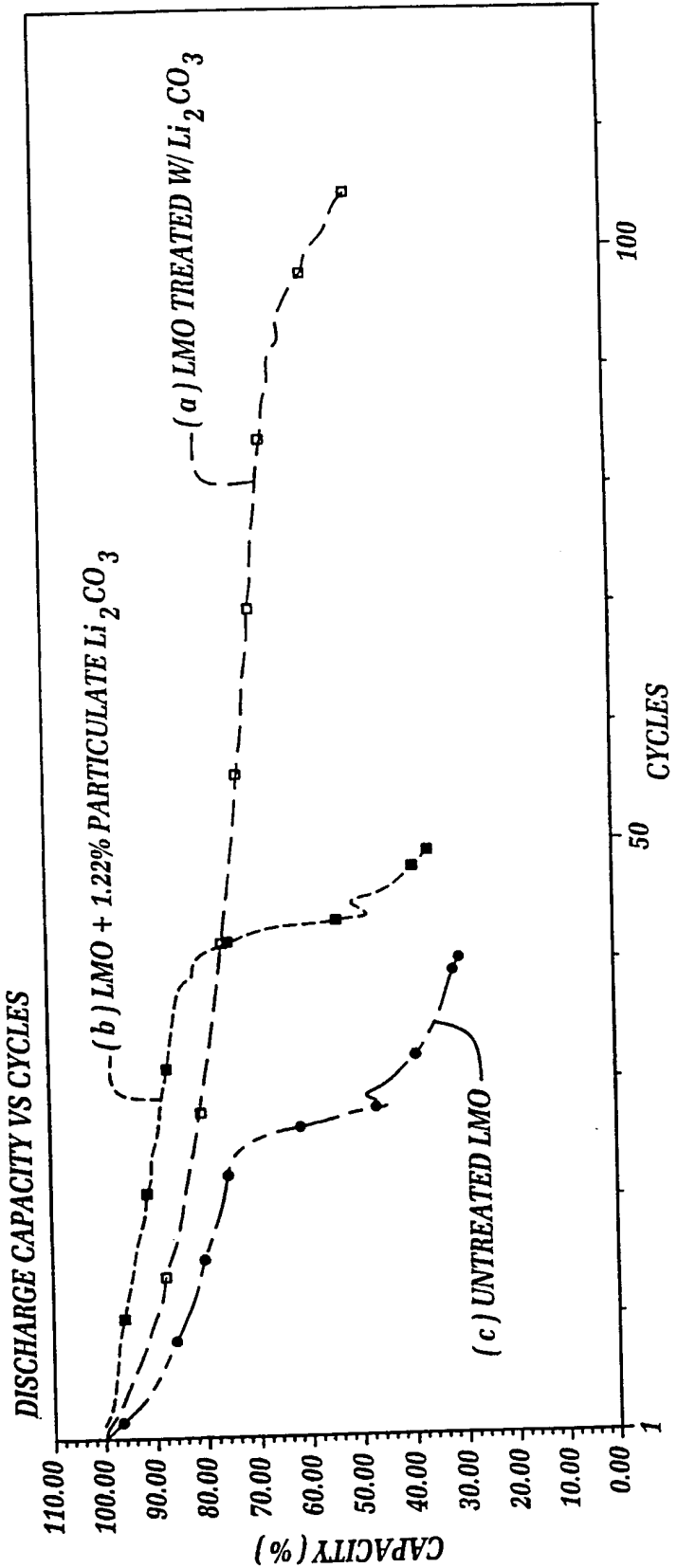


Figure 5B

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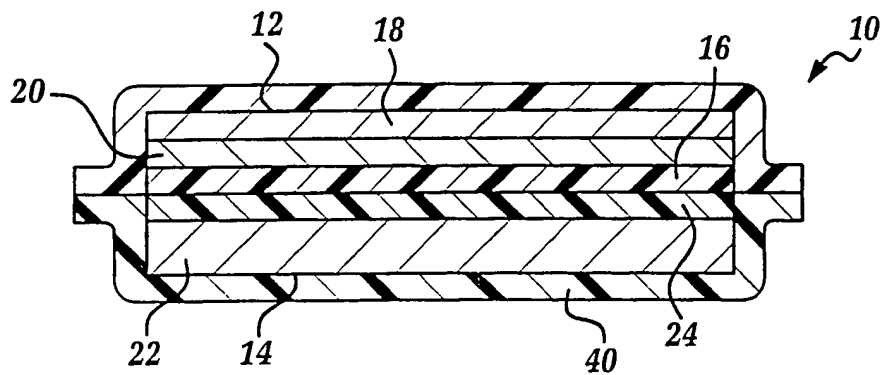


Figure 6

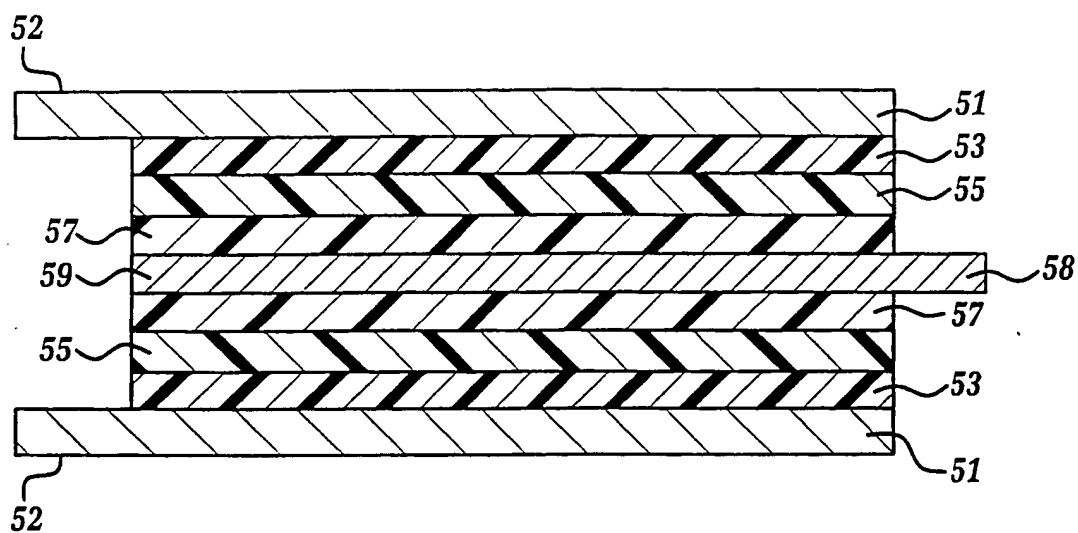


Figure 7